December 19, 1999 (Rev 05/26/2000)

Subject

Instructions for completing Forms pertaining to the submittal of Analytical Data to SCDHEC.

Introduction

These forms are to assist the regulated community in determining what is expected of them in organizing and presenting analytical data to SCDHEC. They ensure that each facility is being treated fairly in that the same type of data is reviewed from each of them. These forms do not request any more information than that which is required to be reported by the SC Certified Laboratory to every customer.

There are 6 Forms and 1 set of instructions. Three forms are applicable to RCRA TCLP, R.261-.270 and are: TCLP Metals, DHEC 3657; TCLP Volatiles, DHEC 3658 and TCLP Semi Volatile, DHEC 3659. Also there are three forms applicable to the "Maximum Contaminates in Drinking Water Regulations" R.61-58.5 (forms: Inorganic, DHEC 3660; Volatile, DHEC 3661; and Semi Volatile, DHEC 3662.

These forms may be used for the submission of analytical results of Waste Stream Characterizations for Industrial Solid Waste Landfills, Soils, Sludges, Remediations and Other Characterizations Submitted to the Department.

The Regulations, Parameters and Analytes are: 1) Industrial RCRA - TCLP Metals, Volatile and Semi-Volatile Organic Compounds; and 2) Industrial - TCLP/R.61-58.5 Inorganic, Volatile and Semi-Volatile Organic Compounds.

Revision Number and Date

Revision Number and Revision Date in the upper right-hand corner should correspond to the most recent data. For example: Revision Number 1, 2, etc. is the second revision, and the date of December 19, 1999 is the date of submission

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of the report to SCDHEC. Please ensure that this reflects the latest (updated) revision number. This revision number and date of revision are necessary to allow a continuous record for each facility waste stream whenever a new waste stream is added to the Industrial TCLP Summary Sheet or a new sample is added to a waste stream.

When adding a new waste stream, simply insert a column and name the waste stream. When adding a sample to a waste stream, insert a column under the waste stream and label it with the sample number and date. A cover letter should explain the purpose of the additional information. Please refer to the paragraph "The Form" for additional information on how to enter the following on the form: sample, more than one sample, future samples, sub-grouping, etc.

The Heading

A) **Revision** of the Forms. A revision is to be documented by a number beginning with the number 1. Whenever a number (1 or > than 1) of samples are added to a waste stream or new waste stream(s) are added to the facility Waste Characterization Study, it is considered revised. When the forms are submitted to South Carolina per regulatory requirements, Department requests or voluntarily by the facility, the latest revision number and the date of submission must be documented. A cover sheet should document all revisions by the facility including the date and to whom they were submitted. For example the cover sheet should be descriptive (Who, What, When, Where and Why).

Type Data: Industrial RCRA - TCLP Metals, Volatile and/or Semi - Volatile Organic Compounds.

The Company (facility) Name: Insert the name of the company. "The **A**ll **B**oot Manufacturing Company"

The Subject/Project: Insert the title assigned to the project. Each project (Landfill, Sludge, Lagoon, Remediation, etc.) is considered a separate project. An example: "Industrial Landfill Permit Request"

The Forms

SCDHEC 3657 (12/99)- Industrial RCRA - TCLP Metals
SCDHEC 3658 (12/99)- Industrial RCRA - TCLP Volatiles
SCDHEC 3659 (12/99)- Industrial RCRA - TCLP Semi-Volatiles
SCDHEC 3660 (11/99)- Industrial Inorganic TCLP/R.61-58.5
SCDHEC 3661 (11/99)- Industrial Volatile TCLP/R.61-58.5
SCDHEC 3562 (11/99)- Industrial Semi-Volatile TCLP/R.61-58.5

1. Waste Stream
Place the name of each waste stream in the columns that scroll horizontally across the page. The width of the Waste Stream column is directly proportional to the number of samples collected for the "Waste Stream". The name of the waste stream can be "Fly Ash (Stack 1), Metal Slag Furnace 1, Lagoon Sludge", etc.

1.1 **Identify the Sample**: The column "Sample 1, Date 1" describes the sample by using text and the date of collection for the sample description. An example: Sample AB, collected 090399 (September 03, 1999). If there is more than one sample collected on the same date for the same waste stream, then use "-1" for the original sample number. For example: use the text "**AB**" (**A**ll **B**oot Manufacturing Company) and the sample collection date 090399, then the "-1" to denote the original sample. That is: AB090399-1, then AB090399-2 for the additional sample.

1.2 **More Than One Sample** Collected On The Same Date. An example of two samples for the same date is: AB090399-1, AB090399-2, etc. Insert a column under Waste Stream 1 or 2, etc., and label it as mentioned in the previous sentence.

1.3 How to **Describe A Sample Sub-Group** of the Original Sample: Label the sub-group with the suffix "-A". For example, AB090399-A represents the first sub-group for the original sample AB090399. AB090399-A1 represents the second sub-group for the original sample AB090399. Placement of sub-groups on the form is simple. Insert two columns adjacent to the original sample AB090399. Label the two columns in sequence as follows: AB090399-A for the first sub-group and AB090399-A1 for the second sub-group. Understand that sub-dividing (sub-grouping) is unusual but is reviewed for clarification. Note that unusual circumstances dictate the use of sub-grouping of a sample.

1.4 **Future Samples for the Same Waste Stream** (IE: Lagoon Sludge) are inserted under the appropriate waste stream. For example, the **A**ll **B**oot Manufacturing Company collects two additional samples on November 1, 1999. Adjacent to the original sample (AB090399) insert two columns for the waste stream Lagoon Sludge. Label them AB110199-1 and AB110199-2. The "original sample" number remains AB090399. Insert the analytical data for the samples under these columns. In summary, Waste Stream 2 columns would now be expanded to include three samples (the "original sample" AB090399, and additional samples AB110199-1 and AB110199-2). NOTE: there is a documentation difference when three (3) samples are collected on the same date (refer

to paragraph 1.2 above) compared with "future samples". The difference is no suffix (-1) assigned the original sample when future samples (at a future date) are to be added. The date of the future samples is different (AB090399 original compared to AB110199-2, -1).

1.5 **Chain of Custody** must be attached for the samples submitted for review.

2. **Facility Sample ID #**
Under the Waste Stream, fill in the Facility Sample Identification Number for the sample. For example, if the **A**ll **B**oot Manufacturing Company is the facility, then use the prefix "**AB**" for the facility. In this example the ID# is AB090399 for Waste Stream 1, Sample 1, Date 1.

3. **Laboratory Sample ID #.** The receiving laboratory fills in the forms or provides the following information to the facility or facility consultant. In this example the ID# is PF090699-1 for receipt of Sample 1, Date 1 for Waste Stream 1.

3.1 **The Laboratory Sample ID Number**. In this case, **P**ine **F**orest Laboratory is the South Carolina Certified laboratory. They received two samples from the same waste stream. The samples were sent by the **A**ll **B**oot manufacturing Company and received by the **P**ine **F**orest Laboratory on September 06, 1999. Therefore, **P**ine **F**orest assigned the sample numbers PF090699-1 and PF090699-2 to describe the laboratory identification (**PF**), sample receipt date (090699) and the sample numbers (1) and (2) from the **A**ll **B**oot Manufacturing Company.

3.2 Future samples and sub-grouping are accomplished in the same manner as mentioned in the paragraph above. Additional clarification can be found under the above section: "Future Samples for the Same Waste Stream" and "Describe a Sample Sub-Group."

Note: One may observe that all sample traceability is maintained from the original sample collection at the facility through the actual analysis of the sample at the certified laboratory.

4) **Laboratory Name** is the name of the laboratory that received the **A**ll **B**oot Manufacturing Company samples. In this example **P**ine **F**orest is the name of the laboratory that received the samples.

5) **Laboratory Certification Number.** The South Carolina DHEC Office of Environmental Certification assigns the **Laboratory Certification Number** to the laboratory. This certification number relates to the specific methods and analytes as certified by the South Carolina DHEC Office of Environmental Laboratory Certification. The laboratory may be fully certified or partially

certified. That is, the laboratory may be certified for specific methods, parameters, and analytes such as TCLP Metals, but not necessarily the TCLP volatile or semi-volatile compounds. This South Carolina Laboratory Certification Number is usually a five-digit number.

6. **Subcontracted Laboratory Certification Number**. This is the number of the South Carolina Certified Laboratory subcontracted for the analytical work.

   6.1 Whenever a subcontracted laboratory generates analytical data, a superscript (#1) is to accompany each analytical value (concentration) for the parameter (analyte) in question. <u>Alternatively</u>, see paragraph 6.3 below for another way to define subcontracted laboratory work for the analytes of concern.

   6.2 For example, Arsenic has a concentration of 2.2 mg/l. This concentration will be denoted on the form as 2.2[1]. Note how footnote #1 is used as a superscript, affixed adjacent to the concentration of the analyte (parameter).

   6.3) <u>Alternatively</u>, the footnote "1" may be placed adjacent to the chemical parameter applicable. For example, refer to the form "Industrial RCRA - TCLP Volatiles then under the subtitle TCLP Volatile Compounds place the number "1" adjacent to this subtitle. The "1" indicates that **all** the volatile organic compounds were analyzed by a subcontracted laboratory. NOTE: no extracted sample can be transferred from one laboratory to another. The laboratory that produces the analytical data must also conduct the QA. A chain of custody must be provided by the contracted laboratory whenever a subcontracted laboratory analyzes samples. That is the change of custody of the samples must be documented on a chain of custody form.

7. **Chain of Custody (COC)** must be attached for the samples submitted for review. The COC is required for the facility sample when it is transferred to the laboratory also when the sample is transferred between laboratories. Should the sample be shipped, a bill of lading must accompany the COC's.

8. **Inorganic TCLP Chemicals**. There are eight RCRA Metals applicable to the TCLP, which are located on the Industrial RCRA - TCLP Form under "analytical parameters." The laboratory or the facility (primary responsibility) should complete the information applicable to these forms. The other two parameters in addition to "Inorganic TCLP Chemicals" are: "Semi-Volatile Organic Compounds" and "TCLP Volatile Organic Compounds".

   8.1 The **Correct Cell to Insert Analytical Date**. There are eight vertical columns adjacent to the analytical parameter column. Fill in the answers horizontally across the sheet under the subjects located at the top of the

vertical columns. For example: The analytical "Digestion Method" is SW3015 for Arsenic, whereas the analytical method is (SW846) 6010B.

8.2 **Find the Correct Cell** to input data: Adjacent to the Blank column, the vertical columns are labeled "Waste Stream 1: Sample 1, Date 1; Waste Stream 2: Sample 1, Date 1," etc. These columns are located at the top of the Industrial RCRA - TCLP Metals, Volatiles, and Semi-Volatiles Summary Sheets. Find the Arsenic (TCLP Metal) parameter located on the left side of the page. Follow it horizontally across the sheet until you intersect the vertical column "Sample 1, Date 1."

8.2.1 For example when using the Industrial RCRA – TCLP Metals form:
1. Locate the Inorganic Parameter Arsenic found on the left side of the page.
2. Locate the vertical column labeled Sample 1, Date 1 found under Waste Stream 1.
3. Follow the Arsenic row horizontally until you intersect the vertical column labeled Sample 1, Date 1 (AB090399).
4. Where the horizontal row for Arsenic and the vertical column for Waste Stream - (Sample 1, Date 1) - (AB090399) intersect represents the cell where you are to place the analytical data for the parameter Arsenic.
5. Insert the concentration for Arsenic in this cell.
6. Continue to fill out the form in this manner. That is, compiling all the data insertions for all the other parameters: Barium, Cadmium, Chromium, Lead, Mercury, Selenium, and Silver.
7. Future Considerations: It is possible that the laboratory can transfer data directly from the analytical instrument to the correct cells. This can be accomplished by an agreement between the Facility and the Laboratory.

9. **Quality Assurance** (for the above samples). Please refer to Paragraph 10, 11 and 12 for the complete synopsis of required QA criteria. All the vertical columns are to be documented with the denotation of Acceptable (A) or Unacceptable (U). For example, an "A" (Acceptable) in the column adjacent to the "Semi-Volatile Extraction Batch Number"(SVEBN) indicates that **all** the criteria for the Batch is acceptable.

9.1 **An example**, the **P**ine **F**orrest Laboratory may use **PF**110199(**A**) for the SVEBN. The **PF** indicates that the laboratory doing the extraction is **P**ine **F**orrest and the suffix **A** indicates the **SVEBN** (sample number **PF**110199) is Acceptable.

9.1.1 Please remember that the **A**cceptability of the SVEBN also means that the following are also Acceptable: The Extraction Blank, Laboratory Control Samples (LCS), Matrix Spike (MS), Matrix Spike

Duplicate (MSD), Unspiked Duplicate (if used), and that the "surrogates" are acceptable.

9.1.2 This Acceptability, under "Semi-Volatile Extraction Records", means all the below criteria are acceptable and completed:

9.1.2.1 Batch Number

9.1.2.1.1 The Date and Time of Extraction

9.1.2.1.2 The analyst

9.1.2.1.3 The EPA Extraction Method Number (for example 3510C)

9.1.2.1.4 The Sample ID of those samples extracted

9.1.2.1.5 The Sample Amount Extracted

9.1.2.1.6 The Extraction Solvent

9.1.2.1.7 The Final Extract Volume.

9.1.2.1.8 Also included are the Standard ID numbers with the amount and concentration added that must be documented on this record.

9.1.2.1.10 Records must also include any cleanup procedures performed on any samples.

9.1.2.1.12 Also, the applicable QA performed for any cleanup procedures must be documented.

9.1.2.1.13 Tractability - It is imperative that Each component of the SVEBN or Volatile Extraction Batch Number (VEBN) above, or any QA on the summary sheets must be traceable to the Specific "Facility Sample and Collection Date". Also the specific paragraph of the paperwork attached to the "Summary Sheets (forms)" are traceable to the specific "**F**acility **S**ample and **C**ollection **D**ate.

Note: One way this may be accomplished is by the use of a systematic index accompanying the attachments to the "Summary Sheets". For example the index sheet should cross-reference the original facility "Waste Stream", "Sample Number/ID" (sample description and date of the sample), the "Laboratory Sample ID Number" (and the chemical parameter applicable such as metals/Volatiles or semi-volatiles) and the analyte name (Arsenic, etc.).

9.2   A checklist is provided in this document to ensure that proper documentations are submitted by the facility along with the Summary Forms. The supporting documentation, mentioned in paragraph 10, 11 and 12 are to be attached for our review. Additionally, the Surrogates (recovery "%" and acceptance - "A") required for the Volatile and Semi-Volatile Analyses are to be documented on the forms. It is suggested the following surrogates be used for recovery.

    9.2.1   Volatile Surrogates- % recovery & **A**cceptability

        9.2.1.1   1,2- Dichlorethane, d4

        9.2.1.2   Toluene, dB

        9.2.1.3   4-Bromofluorobenzene

    9.2.2   Semivolatiles Surrogates- "%" recovery & **A**cceptability

        9.2.2.1   Nitrobenzene, d5

        9.2.2.2   2-Fluorobiphenol

        9.2.2.3   Terphenyl, d14

        9.2.2.4   Phenol, d6

        9.2.2.5   2-Fluorophenol

        9.2.2.6   2,4,6- Tribromophenol

10. **Checklist for TCLP Data Submittals**

    1) **Data on DHEC TCLP Forms.**

    2) **All Applicable Chain-of-Custody Forms.** Ensure that the Chain-of-custody is documented for all laboratories performing analyses.

    3) **Certificates of Analysis for all TCLP Analytical Results.** To include all laboratories reporting TCLP data on the DHEC form. Certificates of analysis must document the laboratory's South Carolina Identification Number, sample identification number, date and time of collection, date and time of extraction (for TCLP and for semi-volatiles), date and time of analysis, EPA method employed for extraction, digestion, sample preparation, EPA method employed for analysis, reporting limit for each method and analyte (based on the low level calibration standard employed in an analysis), results with applicable units, and analyst's initials.

    4) **TCLP Extraction Records for the Bottle Extraction.**

    5) **TCLP Zero Headspace Extraction Records.**

    6) **Metals Digestion Records.**

    7) **Volatile Sample Preparation and Analysis Records.**

    8) **Semi-Volatile Extraction Records.**

11. TCLP Batch Numbers and Pertinent Quality Control

TCLP Batch Numbers for Metals and Pertinent QC

TCLP Bottle Extraction Batch Number   Batch Number given with Acceptable or Not Acceptable
     TCLP Extraction Blank
Digestion Batch Number   Batch Number given with Acceptable or Not Acceptable
     Digestion Blank
     LCS
     Matrix Spike
     Matrix Spike Duplicate
     Unspiked Duplicate (if used)
Analysis Batch Number   Batch Number given with Acceptable or Not Acceptable

TCLP Batch Numbers for Semi-Volatiles and Pertinent QC

TCLP Bottle Extraction Batch Number   Batch Number given with Acceptable or Not Acceptable
     TCLP Extraction Blank
Semi-Volatile Extraction Batch Number   Batch Number given with Acceptable or Not Acceptable
     Extraction Blank
     LCS
     Matrix Spike
     Matrix Spike Duplicate
     Unspiked Duplicate (if used)
     Surrogate Recoveries
Analysis Batch Number   Batch Number given with Acceptable or Not Acceptable

TCLP Batch Numbers for Volatiles and Pertinent QC

TCLP ZHE Extraction Batch Number   Batch Number given with Acceptable or Not Acceptable
     Extraction Blank
Volatile Analysis Batch Number   Batch Number given with Acceptable or Not Acceptable
     Method Blank
     LCS or Calibration Verification
     Matrix Spike
     Matrix Spike Duplicate
     Unspiked Duplicate (if used)
     Surrogate Recoveries

12. <u>Analytical Records to be Submitted for Review</u>

Analytical records from the laboratory performing the analysis must be submitted for each batch number documented on the form.

TCLP Extraction Records for the Bottle Extraction

This record will document the date and time of extraction, analyst, container ID number (to allow the sample containers to be rotated in sequence as the method blank) sample amount extracted and pH checks, and all samples. Quality assurance protocols must include the (pre-test) extraction fluid pH checks, rotation rate checks, and extraction room temperature monitoring. The date, "on" and "off" times and technicians initials must be noted for each process group. All time measurements must be recorded to the closest minute.

TCLP Zero Headspace Extraction Records

These records must include the checks for leaks by either pressurizing the units to 50 PSI for one hour or submerging it in water to check the fittings and rings for any sign of leakage. Notations of these pressure checks must be documented in the records. These records should also include notations for o-ring replacement and piston checks. The extraction fluid ID numbers and records of the ph checks need to be documented in these records along with the ZHE extract device.

The sample temperature adjustment and pH checks, rotation rate checks, extraction room temperature, and on/off times must be noted for each group of samples. Notations for post extraction preservation must also be noted.

Volatiles Sample Preparation and Analysis Records

Volatiles Sample Preparation and Analysis Records will document date and time of analysis, sample preparation procedure (5030B), analyst, instrument ID, amount of sample purged, dilutions, and results. All the samples and pertinent QC processed within this batch must be documented on this record. Samples should be analyzed as soon as possible following the extraction.

Semi-Volatile Extraction Records

The extraction records must be submitted for review to ensure that the pertinent method requirements and quality assurance practices are performed. The records will document the extraction of the TCLP extract and associated QC.

These records must document the semi-volatile extraction batch number, date and time of extraction, analyst, EPA extraction method number (for example, 3510C), sample ID of those samples extracted, sample amount extracted, extraction solvent, and final extract volume. Standard ID numbers with the amount and concentration added must be documented on this record. Records must also include any pertinent cleanup procedures performed on any samples. Applicable QC must be performed for any cleanup procedures used.

13.   Footnotes.
Footnote 1. The subcontract laboratory or the facility uses Footnote 1 to alert us that the analytical data is from a subcontracted laboratory. Using the superscript, place a number 1 (i.e. [1]) in the column adjacent to the Parameter (i.e. Metal, volatile, Semi-Volatile) analyzed by a subcontracted laboratory. For example, in the column adjacent to the gray background colored title "Semi-Volatile Organic Compounds" place the super script number 1 under the appropriate column for the waste stream sample subcontracted to another laboratory. Alternatively, the footnote "1" may be used in the column in lieu of a superscript number 1.